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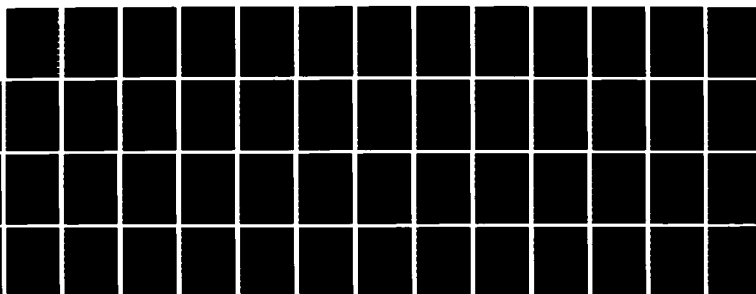
SYMPOSIUM ON APPLIED SURFACE ANALYSIS (7TH) HELD IN
COLLEGE PARK MARYLAND ON 15-17 MAY 1985(U) DAYTON UNIV
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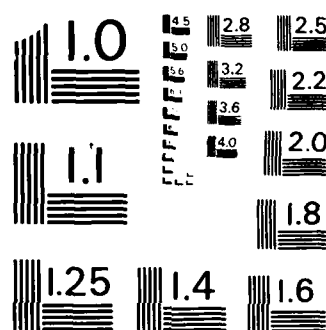
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7th SYMPOSIUM ON APPLIED SURFACE ANALYSIS

by

JOHN T. GRANT

FINAL TECHNICAL REPORT

JULY 1986

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JUL 10 1986
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UNIVERSITY OF DAYTON
RESEARCH INSTITUTE
DAYTON, OHIO 45469

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FOREWORD

This report describes the 7th Symposium on Applied Surface Analysis held at the University of Maryland, College Park Maryland on 15-17 May 1985, under ONR Grant No. N00014-85-G-0118.

The Symposium was conducted by the University of Dayton with Dr. John T. Grant of that organization and Dr. J. S. Murday of the Naval Research Laboratory as Co-Chairmen. This report was submitted in June 1986.

SECTION I INTRODUCTION

The 7th Symposium on Applied Surface Analysis was held at the University of Maryland, 15-17 May 1985. This Symposium was held to meet a need, namely to show the transition between basic surface science research and applications of this research to areas of Department of Defense interest. Areas receiving special attention at this Symposium were: chemical bonding and reactions at metal-semiconductor interfaces; surface analysis and the tribological processes of ion implanted materials; microbeam analysis; and laser ionization of sputtered neutrals. Other topics discussed included adsorption, adhesion, corrosion, wear, and thin films. Approximately 80 scientists active in the field of surface analysis participated in the Symposium. Four scientists presented invited papers at the Symposium. There were 26 contributed presentations. The proceedings of the Symposium have been published in a special issue of the journal "Applied Surface Science" by North-Holland Publishing Company.

Abstracted

/

SECTION II

SYMPOSIUM GOALS

The 7th Symposium on Applied Surface Analysis was held at the University of Maryland, 15-17 May 1985. This Symposium was organized to meet a need, namely, to show the continuing transition between basic research and applications of this research to areas of Department of Defense interest. Workers engaged in basic research and those engaged in applications of surface science research were brought together at the symposium in an environment suitable for promoting the maximum possible interaction between such workers.

The symposium was planned and organized by the University of Dayton with Dr. John T. Grant of the University of Dayton and Dr. J. S. Murday of the Naval Research Laboratory as Co-Chairmen. Four invited speakers were selected to cover specific areas of interest. These speakers and their topics were:

1. John T. Grant
University of Dayton
"Quantitative Depth Profiling"
2. Barbara J. Garrison
The Pennsylvania State University
"Mechanisms of Sputtering"
3. Guy D. Davis
Martin Marietta Corp.
"Adhesive Bonding of Aluminum"
4. Douglas L. Jones
Tektronix, Inc.
"Failure Analysis of Electronic Devices"

Contributed papers on applied surface analysis were also solicited from the research community, resulting in 26 such papers being presented at the symposium.

It was decided to publish as many of the papers as possible in an appropriate journal, thereby providing a permanent record of the work presented at the symposium. The journal "Applied Surface Science" was selected, and the journal editor was pleased to be able to publish such a proceedings. Ten manuscripts were submitted for peer review and publication in the proceedings. The proceedings have been published and distributed to all attendees.

The symposium was advertised through appropriate scientific societies, equipment manufacturers, scientific journals, and at other meetings. It was decided to hold the symposium on the University of Maryland campus and to encourage attendees to stay in the University accommodations in order to provide an informal atmosphere and to optimize the interaction between attendees.

A two-day short course on "Techniques for Surface Analysis," was organized by the University and held in conjunction with the symposium. It was held immediately before the symposium to provide an opportunity for attendees to learn the fundamentals of several surface analysis techniques, so that they could gain as much as possible from the symposium presentations and in their own research.

SECTION III
PARTICIPANTS

Approximately 80 scientists active in the field of surface analysis attended the Symposium.

The list of participants follows.

7th SYMPOSIUM ON
APPLIED SURFACE ANALYSIS

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Livermore, CA 94550

SECTION IV

PROGRAM

The technical program was divided into four sessions. Each session started with a presentation by one of the invited speakers.

The program organization and the abstracts of the papers presented at the symposium follow.

7th SYMPOSIUM ON APPLIED SURFACE ANALYSIS

15-17 MAY 1985

UNIVERSITY OF MARYLAND

GENERAL INFORMATION

The 7th Symposium on Applied Surface Analysis is being supported by the Office of Naval Research and the Air Force Office of Scientific Research. The Symposium will provide an opportunity for workers in all areas of surface characterization to meet and discuss applications of surface analysis. Topics include corrosion and oxidation, adhesion, electronic materials, energy conversion, thin films, thermionic emission, catalysis, and measurement techniques. The aim of this Symposium is to attract participants from universities, government, non-profit, and industrial laboratories to promote the maximum interaction between workers in different areas.

HEADQUARTERS

The Symposium headquarters will be the Center of Adult Education on the University of Maryland campus. Emergency telephone messages can be taken at (301) 985-7303 or 985-7300 during the Symposium. All sessions will be held in the Volunteer Firefighters Room on the second floor of the Center.

REGISTRATION

Symposium registration will be held daily on the second floor, outside the Volunteer Firefighters Room beginning at 8:00 a.m. All attendees must be registered.

PROCEEDINGS

If you plan to publish in the proceedings your manuscript (original plus three copies) should be delivered to the Registration Desk during the Symposium.

NO VENDORS EXHIBITS

No vendors exhibits are being held this year.

LIMOUSINE SERVICE

Limousine service to the Center of Adult Education is available from Baltimore Washington International Airport. This service is provided by Airway Limousine and is \$13 for one person and \$20 for 2-4 persons. The telephone number is (301) 441-2345. Direct taxi fare from Baltimore Washington International Airport to the Center of Adult Education is approximately \$25. Direct taxi fare from Washington National Airport to the Center of Adult Education is approximately \$30. Direct taxi fare from Dulles Airport to the Center is approximately \$35.

ATHLETIC FACILITIES

Any attendee who is staying at the Center of Adult Education may obtain a pass to use the athletic facilities located on the University of Maryland campus by inquiring at the front desk.

LABORATORY TOURS

Lab tours of the University of Maryland, the National Bureau of Standards, and the Naval Research Laboratory are planned for Thursday afternoon (16 May). Please sign up at the Registration Desk. Foreign citizens must have obtained security clearances in advance to visit the Naval Research Laboratory.

LUNCHES

Catered lunches will be served in the Founders Room for those who paid for catered lunches in their preregistration fee.

RECEPTION

A reception will be held in the Chesapeake Room at approximately 5:00 p.m. on Thursday, May 16 following the lab tours.

ASTM MEETING

ASTM Subcommittee meetings on Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy will be held on Friday, May 17 at approximately 1:15 p.m. to discuss the recent ballot on specimen handling, as well as other documents in preparation.

SYMPOSIUM ADMINISTRATION

* * * * *

SYMPOSIUM SUPPORT

Office of Naval Research
Arlington, VA 22217

Air Force Office of Scientific Research
Bolling Air Force Base, DC 20332

SYMPOSIUM CHAIRMEN

James S. Murday
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John T. Grant
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SYMPOSIUM SECRETARY

Alyce Boone
Naval Research Laboratory
Washington, DC 20375

AGENDA

WEDNESDAY MORNING, 15 MAY 1985

8:00 a.m. Registration, 2nd Floor, Adult Education Center

8:50 a.m. Introductory Remarks

SESSION A

Chairman: D. L. Jones
Tektronix
Beaverton, OR

- A-1 9:00 a.m. QUANTITATIVE DEPTH PROFILING
J. T. Grant (Invited)
University of Dayton
Dayton, OH
- A-2 9:50 a.m. PRACTICAL CONSIDERATIONS IN IMPLEMENTING A FACTOR
ANALYSIS APPROACH TO AUGER DEPTH PROFILING
Stephen W. Gaarenstroom
General Motors Research Laboratories
Warren, MI
- 10:10 a.m. COFFEE BREAK
- A-3 10:40 a.m. QUANTITATIVE AES COMPOSITION-DEPTH PROFILES OF THIN FILMS
R. P. Frankenthal and D. J. Siconolfi
AT&T Bell Laboratories
Murray Hill, NJ
- A-4 11:00 a.m. QUANTITATIVE ANALYSIS OF MIXTURES BY AUGER ELECTRON
SPECTROSCOPY
N. H. Turner
Naval Research Laboratory
Washington, DC
and
W. W. Lee
Naval Surface Weapons Center
Silver Spring, MD
- A-5 11:20 a.m. CHARACTERIZATION OF THE IMAGING PROPERTIES OF A
DOUBLE-PASS CYLINDRICAL-MIRROR ANALYZER
N. E. Erickson and C. J. Powell
National Bureau of Standards
Gaithersburg, MD

A-6 11:40 a.m. SURFACE PHENOMENA AT THERMIONIC ENERGY CONVERTER
ELECTRODES
Paul R. Davis
Oregon Graduate Center
Beaverton, OR

12:00 noon LUNCH

WEDNESDAY AFTERNOON, 15 MAY 1985

SESSION B

Chairman: J. D. Venables
Martin-Marietta Corp.
Baltimore, MD

- B-1 1:30 p.m. MECHANISMS OF SPUTTERING
Barbara J. Garrison (Invited)
The Pennsylvania State University
University Park, PA
- B-2 2:20 p.m. AUGER ELECTRON SPECTROSCOPY APPLIED TO THE STUDY OF
CHEMICAL REACTIONS OCCURRING DURING ION IMPLANTATION
D. A. Baldwin, B. D. Sartwell, and I. L. Singer
Naval Research Laboratory
Washington, D.C.
- B-3 2:40 p.m. STOICHIOMETRIC CHANGES IN THE SURFACE OF (100) CUBIC
SiC CAUSED BY ION BOMBARDMENT AND ANNEALING
J. J. Bellina, Jr.
St. Mary's College
Notre Dame, IN
and
M. V. Zeller
University of Notre Dame
Notre Dame, IN
- 3:00 p.m. COFFEE BREAK
- B-4 3:30 p.m. CHEMICAL AND PHYSICAL EFFECTS OF TRANSITION METAL ION
IMPLANTATION INTO COVALENT POLYMERS
P. E. Pehrsson¹, D. C. Weber², and D. E. Ramaker³
¹Catholic University, Washington, DC
²Naval Research Laboratory, Washington, DC
³George Washington University, Washington, DC
- B-5 3:50 p.m. HYDROGEN PROFILING BY ENERGETIC ION BEAM ELASTIC
RECOIL DETECTION
C. R. Gossett
Naval Research Laboratory
Washington, DC

- B-6 4:10 p.m. A SIMS STUDY OF RADIATION INDUCED MIGRATION OF
NITROGEN IN THERMALLY OXIDIZED SILICON
S. A. Schwarz
Bell Communications Research
Murray Hill, NJ
- B-7 4:30 p.m. APPLIED SURFACE ANALYSIS OF Li/SO₂ BATTERY ELECTRODES
Charles R. Anderson
Naval Surface Weapons Center
Silver Spring, MD
- B-8 4:50 p.m. XPS SHAKE-UP STRUCTURE AND SENSITIVITY OF SOME
EXPLOSIVES
J. Sharma, F. Santiago, and M. Chaykovsky
Naval Surface Weapons Center
Silver Spring, MD
- 5:10 p.m. CLOSE

THURSDAY MORNING, 16 MAY 1985

8:00 a.m. Registration, 2nd Floor, Adult Education Center

SESSION C

Chairman: B. J. Garrison
The Pennsylvania State University
University Park, PA

- C-1 8:30 a.m. ADHESIVE BONDING OF ALUMINUM

Guy D. Davis (Invited)
Martin Marietta Corporation
Baltimore, MD
- C-2 9:20 a.m. STUDY OF FIBER FP/MAGNESIUM AND FIBER FP/ALUMINUM METAL
MATRIX COMPOSITES USING IMAGING SECONDARY ION MASS
SPECTROMETRY
Martin A. Rudat and James C. Romine
E. I. du Pont de Nemours and Co., Inc.
Wilmington, DE
- C-3 9:40 a.m. MATERIAL DISPLACING PROCESSES AND SURFACE TEXTURE
Richard B. Zipin
Sheffield Measurement Division, Warner & Swasey Co.
Dayton, OH
- 10:00 a.m. COFFEE BREAK

- C-4 10:30 a.m. RAMAN SPECTROSCOPY OF ADSORBED MOLECULES
D. P. DiLella and R. R. Smardzewski
Naval Research Laboratory
Washington, D.C.
- C-5 10:50 a.m. INFRARED REFLECTANCE SPECTROSCOPY OF SURFACE FILMS
FORMED DURING HIGH TEMPERATURE OXIDATION
D. K. Ottesen and R. W. Bradshaw
Sandia National Laboratories
Livermore, CA
- C-6 11:10 a.m. APPLICATIONS OF SURFACE TECHNIQUES TO THE AGING AND
CORROSION PROBLEMS OF FUZE ENERGIZERS
J. Sharma, F. Santiago, and M. Norr
Naval Surface Weapons Center
Silver Spring, MD
- C-7 11:30 a.m. SURFACE ANALYTICAL INVESTIGATION OF THE CORROSION
BEHAVIOR OF Ti(Pd) SAMPLES
G. Pfennig, H. Moers, H. Klewe-Nebenius, R. Kaufmann,
and H. J. Ache
Institut für Radiochemie
Karlsruhe, FRG
- C-8 11:50 a.m. THIN PLATINUM FILMS AS MODEL ALKANE REFORMING
CATALYSTS
M. J. Wax, R. D. Kelley, and T. E. Madey
National Bureau of Standards
Gaithersburg, MD
- 12:10 p.m. LUNCH
- Afternoon Laboratory Tours
- 5:00 p.m. INFORMAL RECEPTION, Chesapeake Room

FRIDAY MORNING, 17 MAY 1985

8:00 a.m. Registration, 2nd Floor, Adult Education Center

SESSION D

Chairman: John T. Grant
University of Dayton
Dayton, OH

- D-1 8:30 a.m. FAILURE ANALYSIS OF ELECTRONIC DEVICES
Douglas L. Jones (Invited)
Tektronix, Inc.
Beaverton, OR
- D-2 9:20 a.m. GaAs CONTACT METALLIZATION BY ION BEAM MIXING OF
Au-Ge FILMS
M. H. Rashid, A. K. Rai, A. Ezis, and R. S. Bhattacharya
Universal Energy Systems, Inc.
Dayton, OH
- D-3 9:40 a.m. FORMATION OF A SILICON-CARBIDE LAYER DURING CF_4/H_2
REACTIVE ION ETCHING OF Si
G. J. Coyle, Jr.
IBM GTD Poughkeepsie
Poughkeepsie, NY
and
G. S. Oehrlein
IBM Thomas J. Watson Research Center
Yorktown Heights, NY
- 10:00 a.m. COFFEE BREAK
- D-4 10:30 a.m. SPECIAL TECHNIQUES FOR AUGER ANALYSIS OF MICROELECTRONIC
DEVICES
M. S. Leung and G. W. Stupian
The Aerospace Corporation
El Segundo, CA
- D-5 10:50 a.m. GROWTH AND REACTION OF Ti ON Si(111) BY ELECTRON-
EXCITED EXTENDED FINE STRUCTURE
Yves U. Idzerda, Ellen D. Williams, Theodore L. Einstein,
and Robert L. Park
University of Maryland
College Park, MD
- D-6 11:10 a.m. CHARACTERIZATION OF WINCHESTER DISK LUBRICANTS USING
ANGLE RESOLVED ESCA
J. F. Moulder, J. S. Hammond, and K. L. Smith
Perkin-Elmer, Physical Electronics Div.
Eden Prairie, MN

- D-7 11:30 a.m. AN EVALUATION OF THE BREMSSTRAHLUNG CONTRIBUTION TO
SPECIMEN DAMAGE IN XPS USING AUGER PEAK INTENSITIES
M. F. Koenig and J. T. Grant
Research Institute, University of Dayton
Dayton, OH
- D-8 11:50 a.m. SCATTERING OF VERY LOW ENERGY (<100 eV) Ne⁺ IONS
FROM COPPER
Luis L. Tongson
The Pennsylvania State University
Worthington Scranton Campus
Dunmore, PA
and
C. B. Cooper and B. L. Hurst
University of Delaware
Newark, DE
- 12:10 p.m. SYMPOSIUM CLOSE
- 1:15 p.m. ASTM Subcommittee meetings on Auger Electron Spectroscopy
and X-ray Photoelectron Spectroscopy.
The subcommittees will discuss the recent ballot on
Specimen Handling as well as other documents in
preparation.

QUANTITATIVE DEPTH PROFILING

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Depth profiles determine the chemical composition and atomic concentration of solids as a function of distance from the surface. They may be obtained using nondestructive or destructive techniques. The principal nondestructive technique is Rutherford backscattering spectroscopy (RBS) although some information can also be obtained using x-ray photoelectron spectroscopy (XPS) or Auger electron spectroscopy (AES). Destructive techniques include secondary ion mass spectroscopy (SIMS) where emitted ions are measured, and ion sputtering in conjunction with AES or XPS where the remaining surface atoms are measured. Other approaches are crater edge profiling, angle lapping, and ball cratering. Methods to quantify depth profiles will be reviewed.

Wednesday Morning - 9:00 a.m.

PRACTICAL CONSIDERATIONS IN IMPLEMENTING
A FACTOR ANALYSIS APPROACH TO
AUGER DEPTH PROFILING

By

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ABSTRACT

Factor analysis is a data analysis technique which can exploit the Auger line shape information from an Auger depth profile for improved chemical analysis of surfaces. This enables the analyst to determine the number of independent chemical components in a specimen, construct chemical state depth profiles, resolve components with overlapping Auger spectra, extract lineshapes of unidentified components from overlapping spectra, and improve accuracy, precision, and detection limits in quantitative depth profiling.

This presentation focuses on the practical considerations for the analyst interested in implementing factor analysis. Considerations include computer size and speed, software requirements and availability, size of the spectral data matrix, spectral background suppression, and ion beam artifacts.

Wednesday Morning - 9:50 a.m.

- QUANTITATIVE AES COMPOSITION-DEPTH PROFILES
OF THIN FILMS

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ABSTRACT

A Technique utilizing a layer by layer analysis has been developed for obtaining the composition-depth profiles of thin films, e.g. passivating or oxide films, from Auger electron spectroscopy (AES) combined with ion sputtering. The technique, which has monolayer resolution and gives significantly more accurate results than prior methods, has been used for analyzing the composition of the air-formed oxide film on a series of iron-chromium alloys. The new results differ significantly from those that do not consider layer by layer variations in composition, and they are in good agreement with those obtained some years ago by ion-scattering spectrometry (ISS).

Wednesday Morning - 10:40 a.m.

QUANTITATIVE ANALYSIS OF MIXTURES BY AUGER ELECTRON SPECTROSCOPY. N. H. Turner, Surface Chemistry Branch, Naval Research Laboratory, Washington, DC 20375, and W.W. Lee, Materials Division, Naval Surface Weapons Center, Silver Spring, MD 20910

In numerous investigations Auger Electron Spectroscopy (AES) has been used for the quantitative analysis of surfaces. Several different methods of treating the data have been employed; however the most used procedure is the measurement of the peak-to-peak height of the observed derivative AES spectrum vs. a set of standard spectra. In certain cases this method of data analysis has serious shortcomings, e.g. peaks may overlap, or chemical effects have been ignored. Other methods have been suggested, but they have not been used widely. In this study four different procedures for the treatment of AES data for quantitative analysis have been investigated. Mixtures of Ni(OH)_2 and Co(OH)_2 produced by electrodeposition were considered. The AES LMM spectral region of Ni and Co were used, since several of these lines overlap for these elements. The methods tested were the measurement of the peak-to-peak height and of the negative excursion to the baseline intensity. The third procedure used a linear addition of the AES lineshapes of the individual components that is least-square minimized. The last method employed the principal component analysis of a data matrix that relies upon the differences in the AES lineshapes of the individual components. Preliminary analysis indicated some disagreement between the various procedures. The advantages and disadvantages of these different methods will be evaluated, and the need for background subtraction also will be considered for this system.

Wednesday Morning - 11:00 a.m.

Characterization of the Imaging Properties of a Double-Pass

Cylindrical-Mirror Analyzer

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The imaging properties of a commercial double-pass cylindrical-mirror analyzer have been characterized using an extension of the method reported by Seah and Mathieu (1). The electron beam from the coaxial electron gun was rastered across a test surface and the intensity of elastically scattered electrons detected by the analyzer was stored in a computer as a function of beam position on the sample and other experimental parameters. The intensity data could be later plotted to provide an "image" of the detected intensity from the sample. In addition, the elastic peak intensity could be used to modulate the intensity of an oscilloscope, rastered in phase with the beam on the specimen, and the display photographed (1).

Images have been recorded for selected combinations of the following experimental conditions: (a) with the sample aligned using the conventional elastic-peak method and with the sample displaced from that position by up to ± 3 mm along the analyzer axis; (b) for electron beam energies of 100, 500, and 1000 eV; (c) for pass energies of 25, 50, and 100 eV; (d) the selection of small or large analyzer apertures intended for AES or XPS measurements, respectively; and (e) for selected analysis energies ranging from the incident energy, to 1.5 eV above that energy, and up to 50 eV below that energy. Images have also been recorded of the absorbed current to ensure that structure in the elastic-peak data was not dominated by specimen-topography effects.

These detailed measurements shows that this analyzer functions as expected from design considerations. Specimen misalignments, however, have a dramatic influence on the analyzer response and should be avoided when quantitative analyses of selected specimen areas are desired. Examples are given below of images obtained when the specimen was aligned and badly misaligned.

1. M. P. Seah and H. J. Mathieu, Rev. Sci. Instr. (in press).

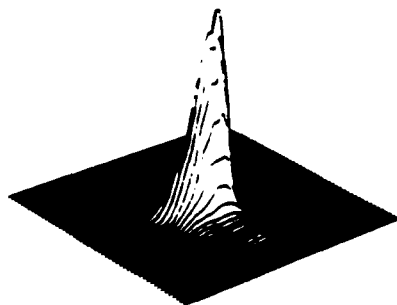


Fig. 1 Image obtained with specimen aligned on analyzer axis; electron energy = 100 eV, horizontal sweep = 13 mm.

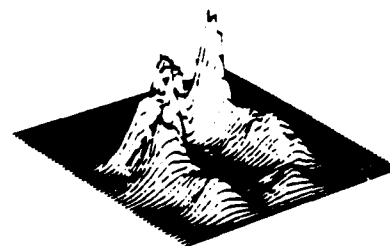


Fig. 2. As for Fig. 1 except that the specimen has been moved 3 mm away from analyzer. Intensities in Figs. 1 and 2 are on different scales.

Wednesday Morning - 11:20 a.m.

Surface Phenomena at Thermionic Energy Converter Electrodes

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Electrode surface phenomena play a variety of roles in the operation of a thermionic energy converter (TEC). Electron emission and electron collection depend upon the work functions of the emitter and collector surfaces, respectively. The work functions, in turn, are fundamental properties of the electrode materials and are strongly affected by the interactions of these surfaces with Cs, O₂, and contaminant gases in the converter. The stabilities of electrode surfaces at operating temperature are affected by evaporation, chemical reactions and faceting.

A typical TEC cesium diode requires an emitted electron current density of about 10 A/cm², at a maximum emitter temperature of 1600-1800 K for efficient practical operation. This implies an emitter with an effective thermionic work function of 2.4-2.7 eV. The traditional TEC uses polycrystalline W or Re as the emitter material, so a cesium pressure of about 1 torr must be maintained to keep the emitter work function at the required value. This requirement introduces losses in output power far greater than would be incurred with only enough Cs vapor present to minimize electron space charge and maintain a sufficiently low collector work function.

TEC design flexibility would be enhanced significantly by the use of emitter materials with inherently low work functions without adsorbed Cs. LaB₆ is one of the few materials which exhibits work functions in the range 2.4-2.7 eV and which is thermally stable at 1800 K. Studies of clean surfaces of this material have demonstrated a significant work function anisotropy with crystal plane and have pointed out the importance of bulk stoichiometry on emission properties and thermal stability.

The (310) face of LaB₆, fabricated with a precise stoichiometry of B/La = 6.09, exhibits an effective work function of 2.4 eV and evaporates less than 10 μm of material per year at 1650 K in vacuum. When heated in > 10⁻⁷ torr of O₂, LaB₆ evaporates at an enhanced rate and faceting occurs. The properties of clean LaB₆ single crystal surfaces and their interactions with O₂ at elevated temperatures will be discussed.

Wednesday Morning - 11:40 a.m.

MECHANISMS OF SPUTTERING

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A classical dynamics model is used to investigate nuclear motion in solids due to bombardment by energetic atoms and ions. Of interest are the mechanisms of ejection and cluster formation both of elemental species such as Ni_n and Ar_n and molecular species where we have predicted intact ejection of benzene- C_6H_6 , pyridine- $\text{C}_5\text{H}_5\text{N}$, naphthalene- C_{10}H_8 , biphenyl- $\text{C}_{12}\text{H}_{10}$ and coronene- $\text{C}_{24}\text{H}_{12}$. The results presented here show that the energy distributions of the parent molecular species, e.g. benzene, are narrower than those of atomic species, even though the ejection processes in both cases arise from energetic nuclear collisions. The bonding geometry also influences the ejection yield and angular distribution. The specific case of π -bonded and σ -bonded pyridine on a metal surface is discussed with comparisons between the calculated results and experimental data. These calculations provide a means of interpreting SIMS and FABMS experimental data. A short computer generated movie will be shown.

Wednesday Afternoon - 1:30 p.m.

AUGER ELECTRON SPECTROSCOPY APPLIED TO THE STUDY
OF CHEMICAL REACTIONS OCCURRING DURING ION IMPLANTATION

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Auger electron spectroscopy has been used to study both the surface reactions and composition of the modified layer resulting from Ti^+ implantation into iron in the presence of reactive gases. It had been previously determined that the beneficial tribological properties imparted by the implantation of titanium ions into various steels is due, at least in part, to the incorporation of carbon as carbides into the implanted layer via chemisorption of residual carbon-containing molecules present in the high-vacuum implanter target chamber. Therefore, it was necessary to obtain, in a more controlled environment, a better understanding of the gas/surface interactions that occur during Ti^+ implantation. The instrument set up for this work, which is described in detail, allows both mechanistic studies via in situ AES monitoring of the Fe surface in a UHV chamber during Ti^+ implantation and compositional analysis via Auger sputter depth profiling. The computer controlled AES allows rapid repetitive acquisition of both lineshape and intensity information as a function of time during implantation and/or reactive gas exposure. For the studies to be reported here, AES analysis was performed with the chamber backfilled with CO and C_2H_2 to study the carburization phenomenon and also with NH_3 to determine if nitridation occurs. At gas pressures of $\sim 10^{-6}$ torr, both molecularly adsorbed and nitride/carbide-like species co-exist on the surface. The nitride/carbide component concentration increases with Ti^+ implant fluence, and the increase is proportional to the sputter-exposed Ti surface concentration $[\text{Ti}]_s$. When the Fe is held at 77K during implantation, the fraction of molecularly adsorbed species as well as the total amount of adsorbate increases, probably due to an increased gas molecule sticking probability. At any constant $[\text{Ti}]_s$ value there is a steady-state adsorbate concentration due to competition between adsorption and sputtering by Ti ions. The $[\text{Ti}]_s$ increases during the implantation, increasing the adsorbate steady-state concentration. Kinetic equations for the adsorption/sputtering have been developed and solved to generate a mathematical model that compares favorably with the AES concentration versus ion fluence measurements.

Wednesday Afternoon - 2:20 p.m.

Stoichiometric Changes in the Surface of (100) Cubic SiC
Caused by Ion Bombardment and Annealing

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and

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The (100) surface of cubic silicon carbide has been studied by Auger Electron Spectroscopy as a function of argon ion damage and in-situ annealing in ultra-high vacuum. The results indicate that ion bombardment systematically increased the Si/C peak height ratio with increasing ion beam energy above 500 eV, although the reported sputtering yield for bulk silicon is higher than that for carbon. The line-shape fine structure of both the silicon and carbon peaks was destroyed by ion bombardment but readily recovered by a brief heat treatment at about 800 degrees Celsius. Further heating above about 1000 Celsius monotonically decreased the Si/C ratio with increasing temperature. Comparison of the silicon and carbon Auger peak height changes with that from imbedded argon show that this decrease in Si/C ratio was caused by silicon depletion rather than by carbon accumulation on the surface. A systematic study of the changes in the silicon and carbon Auger peak heights as a function of time and temperature showed that the depletion occurred with an activation energy of about 100 kcal/mol (4 eV/atom). The carbon Auger line shape indicates that as the silicon was desorbed, the carbon bonding eventually changed from carbidic to graphitic.

Wednesday Afternoon - 2:40 p.m.

CHEMICAL AND PHYSICAL EFFECTS OF TRANSITION METAL ION IMPLANTATION INTO COVALENT POLYMERS

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D.C. Weber, Code 6127, Chem. Div., Naval Res. Lab, Wash. D.C.,
20375

D.E. Ramaker, Chem. Dept., George Wash. Univ., Wash. D.C., 20052

This work examines the effects of implantation on graphite and a variety of polymers including polyacetylene, polyethylene, and poly(vinylidene fluoride). Other workers have shown that halogen implantation into polyacetylene (1) or poly(phenylene sulfide) (2) results in large conductivity increases and greatly enhanced resistance to air oxidation.

The rapid introduction of large amounts of energy during implantation causes significant damage to the pristine film. We have utilized surface science techniques to obtain information about the effects of ion beams on the fragile, covalent polymeric structure, and also on the post-implantation metal-matrix and metal-metal chemical interactions. Specific chemistries were investigated in view of the potential bonding sites in the original films. Auger carbon KLL lineshapes are used to differentiate various carbon forms, e.g., amorphous carbon, graphite, i-carbon, or carbide. Characteristic Electron Loss Spectra corroborate the Auger results. Both X-Ray Photoelectron Spectroscopy (XPS) and Auger confirm that implantation radically alters the original polymer structure and stoichiometry, and ultimately yields amorphous carbon. XPS is also used to ascertain implant oxidation states, and the degree of metal dispersion or precipitation in the carbon matrix. High dose iron implants undergo a transition from small aggregates to bulk metal clusters. These larger metal clusters appear to cause regraphitization of the surrounding carbon.

Conversion Electron Mossbauer Spectroscopy (CEMS) is used to characterize implanted iron, its aggregation, and possible complexation with unsaturated or graphitic bonds.

*Work funded by Office of Naval Research, Contract N00014-84-K-2006

Wednesday Afternoon - 3:30 p.m.

HYDROGEN PROFILING BY ENERGETIC ION BEAM ELASTIC RECOIL DETECTION

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Two methods of profiling hydrogen in the near-surface region by means of energetic ion beams are in use with the NRL Van de Graaff. In both cases a 3 MeV $^4\text{He}^+$ ion beam is used to produce by elastic scattering the recoils of hydrogen from the near surface region of the sample. The lower depth resolution method utilizes a surface barrier detector placed at 25° to the beam and covered with a 5 μm Ni foil, which is sufficiently thick to stop the scattered ^4He ions but still transmit the recoil H ions at reduced energy. The higher resolution method utilizes a double focusing magnetic spectrometer placed at 45° to the beam with 2% energy acceptance on a focal-plane position-sensitive detector. In this case discrimination of the recoiling H^+ ions from the scattered $^4\text{He}^{2+}$ ions is provided by the different energy loss of the two ion species in the surface dead layer of the position-sensitive detector. The two distributions are sufficiently separated in the energy spectrum from the detector to allow placing a window over the H distribution. The resulting gate is applied to the position signals from this detector producing a position, or momentum, distribution of the H ions only, from which the hydrogen profile is determined. Examples of the application of these methods to samples from ion implantation, deposition and ion beam enhanced deposition will be shown. Our experience shows that hydrogen incorporation in such samples is more prevalent than previously believed, but the lack of easily applied methods of profiling H has previously hampered such determination.

Wednesday Afternoon - 3:50 p.m.

**- A SIMS Study of Radiation Induced Migration of Nitrogen
in Thermally Oxidized Silicon**

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ABSTRACT

The potential use of thin silicon nitroxide films as gate dielectrics in VLSI MOS devices has motivated much recent work. The present study shows that positive ion bombardment, as encountered in sputter depth profiling or ion implantation, can induce considerable movement of nitrogen in thin thermal oxide films on silicon. Low Energy N_2^+ (5.2 kV) implants were performed in-situ in a Cameca IMS-3f SIMS (secondary ion mass spectrometry) apparatus and were subsequently depth profiled. The effects of implant dose and oxide thickness were examined and comparisons made to films prepared by rapid thermal nitridation and LPCVD. Profiles obtained under O_2^+ , O^- , and Cs^+ bombardment were also compared. SIMS depth profiles of 170Å oxides using positive ion bombardment show a depletion of nitrogen near the surface, a shoulder in the nitrogen concentration near the Si-SiO₂ interface, and a peak in this concentration at the interface. This behavior is not observed in samples thicker than 300Å or in samples bombarded by negative ions. No dependence on nitridation method or crystal orientation was observed. The results suggest that ion bombardment creates positively charged nitrogen interstitials which, under the influence of a high electric field in the oxide, migrate rapidly to the Si-SiO₂ interface. The interface acts as a sink for nitrogen at low temperatures.

Wednesday Afternoon - 4:10 p.m.

APPLIED SURFACE ANALYSIS OF Li/SO₂ BATTERY ELECTRODES

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A discussion of the methodology and an overview of the results of an investigation to determine the chemistry of the lithium anodes and carbon current collectors of stored and discharged commercial high-rate lithium/sulfur dioxide primary battery cells are presented. Elimination of the safety hazards of this critically-needed battery is the objective of this highly applied study of a very complex and most un-model system. Correlated XPS and mass spectroscopy analysis of the electrodes as a function of electrode sample temperature proved highly useful. Argon ion sputtered neutral species from both electrodes were mass analyzed and lithium anodes were examined with a scanning Auger microprobe. These analyses were performed for electrodes discharged at a 0.77 mA/cm² rate to a 2.0 V cut-off at temperatures of -30, 23, and 70 C.

Reaction products of nearly every cell component were identified on the electrodes, especially in cells discharged at 70 C. The sulfur chemistry is complex, especially so for these cells, which were stored for several years prior to discharge. This work showed that the passive film on the lithium anode was not always simply lithium dithionite as had been previously believed. The lithium anode chemistry is strongly inhomogeneous both laterally and with depth. Parts of the anode gave evidence of the local action of strong oxidizers. This first and only such study of the carbon current collector yielded several surprises. The only discharge product of the cell has been widely regarded to be lithium dithionite, which is deposited at the carbon electrode. Again, this view was greatly oversimplified. The most remarkable finding was the presence of LiH in quantities of as much as 35% of the lithium deposited at the carbon electrode. The LiH appears to be formed as a graphite intercalation compound, which prevents its immediate oxidation by the SO₂ and allows its accumulation.

Our on-going effort is to make and examine the properties of this LiH graphite intercalation compound. While the LiH intercalation compound has not been otherwise made, both KH and CsH graphite intercalation compounds have been. The recently much-studied KH system acts as a nearly reversible sink or source of hydrogen. If the LiH system has similar properties, it may well be a cause of safety problems. Thus, from myriad possibilities, this initially unforeseen basic issue appears to bear directly on the cell safety.

Wednesday Afternoon - 4:30 p.m.

XPS SHAKE-UP STRUCTURE AND
SENSITIVITY OF SOME EXPLOSIVES

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Polyaminopolynitrobenzenes show pronounced shake-up structure in the XPS spectra of the nitrogen and oxygen atoms of the nitro group. This structure arises from excitation of the molecule during ejection of the photoelectron. We have shown that the shake-up separation in different derivatives of the molecules correlates with the explosive sensitivity. When the energy of the monopole transition is large, the explosive shows large insensitivity. This is the first time that a relationship between these two fast processes (molecular shake-up and explosive initiation) has been shown to exist. In order to understand the nature of the fast shake-up process in triaminotrinitrobenzene (TATB), the dependence of the shake-up has been investigated as a function of the photon excitation energy. For this purpose, the K x-ray lines of Ti (4510.8 eV), Mg (1253.6 eV) and O (524.9 eV) have been used. There is little difference between spectra obtained with Mg and Ti anodes. However, when the oxygen anode was used, an additional shake-up satellite was found at a separation of 5.8 eV from the parent line. The position of the additional satellite coincides with that of one of the energy loss peaks. Thus, it appears that when the escape velocity of the photoelectron decreases from 848 eV to 120 eV, the molecule can be excited to more than one state, even though the excitation is still in the sudden approximation regime. The present result shows that the degree of excitation of the molecule depends upon the photoelectron escape velocity, that is, on the time taken by the electron to get out of the coulombic interaction range of the molecule.

Wednesday Afternoon - 4:50 p.m.

ADHESIVE BONDING OF ALUMINUM

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ABSTRACT

To conserve weight, modern aerospace structures are usually adhesively bonded. This bond must have high initial bond strength and long term bond durability. The former is provided by a microscopically rough Al oxide that allows mechanical interlocking between the adherend and the adhesive primer. The latter is related to resistance to hydration in a moist environment. The resistance is, in turn, governed by the surface chemistry of the adherend. Bonds prepared with phosphoric-acid-anodized (PAA) adherends or Forest Products Laboratory-etched (FPL) adherends pretreated in a solution of an inhibitor, such as nitrilotris methylene phosphonic acid (NTMP), exhibit excellent durability. To understand the mechanisms that make these surfaces so stable, we have examined the inhibitor-adsorption and hydration processes using mechanical testing and surface behavior diagrams (SBD's) to analyze x-ray photoelectron spectroscopy (XPS) data. Adsorption of NTMP onto FPL surfaces displaces the water initially present with the formation of approximately one monolayer of tightly bound inhibitor. Upon exposure of a PAA or an inhibited FPL surface to high humidity and temperature, hydration occurs in three steps: i) reversible physisorption of water, ii) slow dissolution of the surface phosphate or inhibitor-aluminum complex followed by rapid hydration of the freshly exposed amorphous aluminum oxide to boehmite, and iii) further hydration of the boehmite to bayerite. Finally, several criteria important for inhibitors designed to improve bond durability have been identified: i) displacement of water and occupation of all active sites on the Al_2O_3 surface, ii) formation of strong inhibitor surface bonds, iii) insolubility of the resulting inhibitor-aluminum complex in aqueous solutions, iv) compatibility with the adhesive or primer, and v) coupling of the inhibitor to the adhesive.

Thursday Morning - 8:30 a.m.

STUDY OF FIBER FP/MAGNESIUM AND FIBER FP/ALUMINUM METAL MATRIX
COMPOSITES USING IMAGING SECONDARY ION MASS SPECTROMETRY

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Textile Fibers Department
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Wilmington, Delaware 19898

Fiber FP (100% alumina fiber) reinforced metals exhibit dramatically improved properties compared to unreinforced metals which can allow these composites to replace much larger and heavier standard components in high-stiffness and high temperature applications. The transmission of stress to the fibers is important in these systems, and therefore it is of great interest to understand the bonding interfacial zone between the fiber and metal. Differing interpretations of analyses of this zone in FP/magnesium composites exist in the literature, supporting either a magnesium aluminum spinel or a magnesium oxide composition, and little has been reported about the bonding of alumina fibers to aluminum/lithium alloys. We have applied imaging SIMS to these problems because of its sensitivity and chemical information content, and have also explored the distributions of other components present in the metal and compared them with their expected roles.

Transverse and longitudinal sectioning revealed that the interfacial reaction zone is approximately three to four microns in thickness in the Mg samples, and emits ions characteristic of magnesium aluminum spinel when bombarded by energetic ions in the SIMS instrument (based on comparison with an authentic sample). The images also revealed that some magnesium oxide may be present, but we believe this to be a minor component. Alloying elements were found to be inhomogeneously precipitated about the fibers, as might be expected from their roles as crystallization initiators or grain refiners. In samples for which boron nitride was used as a mold-release agent, boron was discovered to have migrated into the interfacial zone.

Therefore, in contrast to recently published work based on scanning Auger microprobe line scans, we believe that the interface is primarily magnesium aluminum spinel, not magnesium oxide.

SIMS is ideally suited to the determination of the fate of lithium in FP/Al-Li composites, due to its extreme sensitivity to all alkali metals. Prior to this work, it was believed, without proof, that lithium is the key ingredient in the interfacial zone that allows wetting of the fiber by aluminum. The images clearly show a lithium-rich reaction zone which has a thickness dependent upon casting conditions, supporting this view. In those samples considered to have the best balance of properties, the zone was determined to be approximately three microns thick. Evidence suggests that this may be an intermetallic (lithium-aluminum) oxide region. Separate experiments to study a possible mechanism of lithium transport utilized SIMS depth profiling of fibers to reveal that the lithium can arrive at the fibers through the gas phase under certain conditions, prior to the arrival of the molten aluminum.

Thursday Morning - 9:20 a.m.

MATERIAL DISPLACING PROCESSES AND SURFACE TEXTURE

by
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Abstract

Surfaces exhibit strata which are characteristic of the processes used to create them. These strata are most readily identified for analysis when the cumulative distribution of the height of the surface profile is plotted on normal probability coordinates. Since the information contained in these plots is a complete description of the height variations of the surface profile, they provide a useful tool for the analysis and specification of the texture of engineering surfaces.

The existence of such strata has been recognized for quite some time [1,2,3], but the theory required to analyze them has only recently been begun to be developed [4]. The most recent publication [5] has presented analyses of the strata created by processes which either add to or remove material from a surface profile. The present work treats the class of processes in which material is neither added to nor removed from the surface profile, but is merely displaced from one region to another within the profile. The specific processes treated are scratching and coining.

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2. J.B.P. Williamson, J. Pullen and R.T. Hunt, The Shape of Solid Surfaces, in: Surface Mechanics, Ed. F.F. Ling (ASME, New York, 1969).
3. J.B.P. Williamson, The Shape of Solid Surfaces, in: Rough Surfaces, Ed. T.R. Thomas (Longman, London, 1982).
4. R.B. Zipin, Appl. Surface Sci. 15 (1983) 334.
5. R.B. Zipin, Appl. Surface Sci. 18 (1984) 123.

Thursday Morning - 9:40 a.m.

RAMAN SPECTROSCOPY OF ADSORBED MOLECULES

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Vibrational spectroscopy yields more specific information on molecular bonding and structure than electronic spectroscopy. Vibrational spectroscopy of adsorbed molecules and monolayers has not been commonly employed because of the the low sensitivities of vibrational techniques. Obtaining the Raman spectrum of a monolayer can be a formidable task since Raman scattering is generally a weak phenomenon. In recent years, however, Raman spectroscopy of adsorbed molecules and monolayers has become more feasible. The most exciting development has been the discovery of surface-enhanced Raman scattering (SERS) which in some cases is as much as a million times stronger than normal Raman scattering. Resonance Raman scattering (RRS) can be nearly as strong as SERS and improvements in instrumentation have greatly expanded the scope of this technique. We report SERS and RRS spectra of adsorbed molecules and monolayers with emphasis on the spectra of Langmuir-Blodgett (LB) films.

The LB films were made using classical film balance techniques. One or more monolayers of a 1:1 tetracumylphenoxy-phthalocyanine:stearyl alcohol mixture were transferred to either a silica, gold, silver or palladium support. The metal supports were prepared by vapor phase deposition of a thick film of metal onto a silica substrate. Metal-free, Cu-, Pt- and Ni-substituted phthalocyanines were studied and good quality spectra were obtained even for single monolayer films. Typical experimental conditions included excitation with 15-100 mw of laser radiation, 90 degree scattering geometry and analysis with a conventional Raman spectrometer using a spectral bandpass of 4-8 wavenumbers.

The vibrational frequencies for molecules in a LB film were usually the same as those observed for the molecule in the solid state suggesting that the environment for the molecules in the film is not too different from the solid state environment. Small differences were sometimes seen and in one case dimerization of the phthalocyanine molecules in the film was indicated.

The Raman spectra of adsorbates on metal surfaces are emphasized. Depending on the geometry of the surface, Raman scattering from molecules adsorbed on a metal may be either stronger or weaker than the scattering by the corresponding free molecules. Fluorescence, which can obscure the observation of RRS, is often selectively quenched relative to RRS near metal surfaces.

For both SERS and RRS not all bands in a spectrum are enhanced equally. The interpretation of the spectra in terms of the selective mode enhancement is discussed.

Thursday Morning - 10:30 a.m.

Abstract submitted to the 7th Symposium on Applied Surface Analysis

INFRARED REFLECTANCE SPECTROSCOPY OF SURFACE FILMS
FORMED DURING HIGH TEMPERATURE OXIDATION*

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ABSTRACT

Fourier transform infrared (FT-IR) reflectance spectroscopy has been developed as a sensitive analytical technique for the characterization of thin non-metallic films on metallic substrates at our laboratory. Our efforts have been directed primarily at understanding the oxidation of metals and ferrous alloys which have been exposed to high temperature environments.

Film thicknesses encountered in our studies ranged from less than 100 angstroms to more than 10 micrometers. Our principal concern has been the interpretation of these spectra. Prior work using this technique has suffered from poor agreement of reflectance measurements with available transmission reference spectra, and the inability to account for changes in reflectivity due to the non-ideal nature of the films.

We will discuss experimental data in the light of our theoretical calculations. Quantitative comparisons will be made for oxide films formed during the high temperature oxidation of chromium and binary iron-chromium alloys. These calculations are based on optical constants for various metal oxides, and the method used to perform these calculations will be briefly described. The calculated sensitivity of this external reflectance technique will be given for principal bands in the spectra of several transition metal oxides.

Experimental data will be presented for the oxidation of stainless steels in air at various temperatures. This results in the formation of very thin films which, according to our calculations, contain a mixture of oxide species. These range from simple chromium and iron oxides to complex Fe/Cr oxide solid solutions and spinel structure phases. Preliminary work will be discussed relating the composition and thickness of the oxide films to temperature and alloy composition.

*Work supported by the U.S. Dept. of Energy, Contract DE-AC04-76DP00789.

Thursday Morning - 10:50 a.m.

APPLICATIONS OF SURFACE TECHNIQUES TO THE
AGING AND CORROSION PROBLEMS OF
FUZE ENERGIZERS

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Naval Surface Weapons Center
Silver Spring, Maryland

A combination of surface analytical techniques such as XPS, SAM, SEM, and EDAX can be very effective in tracking down problems caused by surface reactions and corrosion. This was found to be so during the investigation of artillery fuze energizers, which had shown unexpectedly short shelf-life precipitated by corrosion. The problem caused perforation of the battery walls and its failure while the leaking electrolyte could be a potential hazard to the detonators in the fuzes. Optical and SEM photomicrographs identified the local areas with characteristic hemispherical pits where the droplets of the incompatible liquid had caused corrosion. XPS and EDAX were used to identify the culprit reaction in the background of other harmless products. Methylene bromide, used as an insulating liquid, and believed to be compatible with copper and fluoboric acid during the design of the energizers proved to be the cause of corrosion. Deep inside the corrosion pits, crystals of copper bromide evidenced the corrosive reaction, which had shortened the expected life of the energizers from twenty years to two years. The droplets of methylene bromide, which caused the problem, were being released by the failure of the polymeric sponge in holding the dense liquid during storage and transportation.

Thursday Morning - 11:10 a.m.

Surface Analytical Investigation of the
Corrosion Behavior of Ti(Pd) Samples

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Titanium with an admixture of 0.2wt. % of Palladium is under discussion as material for containers of vitrified highly active waste. The corrosion behavior against brines was investigated in previous laboratory and field experiments. In order to extrapolate to longer standing times, the knowledge of the underlying chemical processes is of importance.

Using the surface analytical techniques XPS and AES we have investigated the composition of the surface and the corrosion layer of Ti(Pd) samples after different corrosion treatments. Depth profiling studies aimed at a determination of the components within the oxide/corrosion layer. The oxidation states of Ti, however, could only be determined within a layer corresponding to the information depth of XPS, since by the sputtering process during depth profiling a strong reduction of higher oxidation states of Ti takes place. Within layers thinner than ~ 5 nm only TiO_2 was observed for oxidized and corroded samples with an upper limit of 10% for admixtures of other oxides. Depth profiles revealed no further significant components besides Ti and O but showed that the interface region between oxide layer and metallic bulk is very inhomogeneous. Sputtering coefficients for 5 keV Argon ions were measured in separate experiments for homogeneous standard samples for both TiO_2 and Ti-metal. We obtained $Y(\text{TiO}_2) = 2.37$ and $Y(\text{Ti-metal}) = 1.62$.

No influence on corrosion layer thickness and composition was found for different corrosion times (67-270 d) and temperatures (90, 170°C) within the experimental accuracy. Application of strong γ -radiation during a corrosion treatment, however, revealed a thick (up to 200 nm) overlayer containing Mg, Si, and O. It has to be studied further whether the Silicon, originating probably from the glass vessel used in this experiment, is primarily responsible for the generation of this overlayer.

Thursday Morning - 11:30 a.m.

THIN PLATINUM FILMS AS MODEL ALKANE REFORMING CATALYSTS
M. J. Wax, R. D. Kelley, and T. E. Madey, Surface Science Division
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Vapor deposition onto a tungsten (100) substrate in ultrahigh vacuum has been used to prepare model supported platinum catalysts which are free of extraneous promoters and poisons. Various platinum loadings (submonolayer to multilayer) have been achieved reproducibly. These materials catalyze alkane reforming at a rate significantly greater than that of the clean tungsten surface. Their selectivities and specific activities will be discussed and contrasted with those of single crystal and supported platinum.

Thursday Morning - 11:50 a.m.

FAILURE ANALYSIS OF ELECTRONIC DEVICES

by

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The electronic device industry has benefited greatly from the growth and application of surface analytical techniques to component failure analysis. High reliability of electronic devices is expected by the user, resulting in greater demands on the designer and manufacturer to overcome failures by understanding causes and eliminating them rather than merely replacing parts in the field. The high technology nature of most electronic components today has required the shift from more conventional bulk analytical techniques of the chemistry laboratory to the surface and near-surface analytical techniques of the current failure analysis and materials characterization laboratories.

Integrated circuit failure analysis through the application of surface techniques is becoming the norm. Novel applications to not only the silicon field, but also the germanium and gallium arsenide device areas will be reviewed. Failures also occur with resistor and capacitor materials which can uniquely be addressed with surface techniques. The field of switches and contacts has numerous examples of failures being understood by means of surface analysis as is true for circuit board and hybrid substrate problems. In the display device industry both cathode ray tube and flat panel displays have been made more reliable as a result of understanding gained from appropriate analyses. Various technique applications will be presented to encourage the scientist and engineer to broaden their scope of potential problems in failure analysis which can beneficially be addressed using surface analytical techniques.

Friday Morning - 8:30 a.m.

GaAs Contact Metallization by Ion Beam Mixing of Au-Ge Films

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We report formation of ohmic contact by ion beam mixing of Au-Ge thin films deposited on GaAs. Alternate layers of Au and Ge films were deposited by the electron beam evaporation technique. The films were irradiated with 1 MeV Au^+ , 300 keV Ge^+ , and 80-140 keV Si^+ ions at various fluences in the range of 1×10^{15} to $2 \times 10^{16} \text{ cm}^{-2}$. Co-deposited Au-Ge layers evaporated from a eutectic melt were also used. Interdiffusion, phase formation and the change in surface morphologies resulting from ion beam mixing have been analyzed by Rutherford backscattering, Auger electron spectroscopy and electron microscopy. Homogeneous alloying and remarkable improvement in surface morphology were achieved due to the ion bombardment. It was observed from cross-sectional TEM that mixing at ion energies high enough to penetrate through the metallization introduced damage into GaAs. Contacts were unstable and non-ohmic in these cases. However, ohmic contact was obtained at lower energies and doses where penetration was not possible and therefore very little defect was created in the interface region of GaAs.

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Friday Morning - 9:20 a.m.

FORMATION OF A SILICON-CARBIDE LAYER DURING CF_4/H_2 REACTIVE ION ETCHING OF SI

G. J. Coyle, Jr. and G. S. Oehrlein*

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The anisotropy of reactive ion etching (RIE) is related to low energy ion bombardment of the material being etched. The low energy ion exposure of electronic materials can result in bombardment damage, which can affect the device applicability of these materials in a detrimental way. Indeed many reports have appeared in the literature, where detrimental side effects of dry etching techniques on semiconductor substrates were reported and related to dry etching induced damage in the semiconductor substrates. In the case of silicon the following effects have been observed: A degradation of the minority carrier lifetime, changes in the barrier height and of the ideality factor of Schottky barriers formed on dry etched Si, high contact resistance in contact hole etching and a deterioration of the oxide quality (interface state density, dielectric strength) of thermal SiO_2 -films grown on dry etched substrates.

In the present paper we report results of a study aimed at identifying the structural and chemical changes of the surface and near-surface properties of Si caused by reactive ion etching. Silicon specimens which had been etched in CF_4 and CF_4/H_2 have been characterized by X-ray photoelectron emission spectroscopy. Angular rotation was used to study films deposited by the plasma process onto the Si surface. An intriguing new finding was the discovery of a silicon carbide layer localized near the fluorocarbon film/Si interface.

Results are summarized as follows: i) Exposure of Si surfaces to CF_4/H_2 RIE produces a silicon carbide layer localized near the deposited fluorocarbon film/Si interface. ii) The existence of this carbide layer appears to be independent of gas composition from 0 to 40% H_2 for a one-minute overetch. iii) In agreement with previous investigations [1,2], the thickness of the C, F-containing film increases as H_2 is added to the CF_4/H_2 plasma but does not exceed 50\AA . The existence of the silicon carbide layer may be significant in understanding the mechanism of fluorocarbon film formation and the selective nature of reactive ion etching as well as the detrimental side-effects of this processing method.

- 1 G. S. Oehrlein, R. M. Tromp, Y. H. Lee, and E. J. Petrillo, Appl. Phys. Lett. **45**, 420 (1984).
- 2 G. S. Oehrlein, G. J. Coyle, Jr., I. Reimanis and Y. H. Lee (to be published).

Friday Morning - 9:40 a.m.

SPECIAL TECHNIQUES FOR AUGER ANALYSIS
OF MICROELECTRONIC DEVICES

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ABSTRACT

Microelectronic devices are becoming more complex and device features are getting smaller as the level of integration continues to increase. Although scanning Auger microscopy has been applied extensively to the analysis of microelectronic devices with a great deal of success, the analysis of current and future devices is presenting new challenges. The major limitations are 1) features of interest in microelectronic circuits are often comparable in size to the beam diameter of commercial Auger microprobes, and 2) the electron beam tends to drift about on the specimen surface because of mechanical instability and differential thermal expansion of the apparatus. In this paper, we present two different techniques developed to overcome these limitations. In the sample modulation technique, the modulating signal is applied to the electrically isolatable regions of a device instead of to the electron energy analyzer. This method of modulation permits the detection of only the Auger electrons that are emitted from the modulated region. Spurious contributions from adjacent areas inadvertently illuminated by the analyzing beam are suppressed. In the position modulation technique, the analyzing beam is scanned repetitively across the feature to be analyzed and the Auger signal is synchronously detected at the scan frequency. The resulting Auger signal magnitude is shown to be unaffected by beam drift. This method of signal detection eliminates the error and uncertainty caused by beam instability during long-term depth profiling.

Friday Morning - 10:30 a.m.

GROWTH AND REACTION OF Ti ON Si(111) BY
ELECTRON-EXCITED EXTENDED FINE STRUCTURE

by

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and Robert L. Park

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Three variations of the electron-excited Extended Fine Structure (EFS) technique, Auger Monitored Extended Fine Structure (AMEFS), Reflected Electron Energy Loss Fine Structure (REELFS), and Extended Appearance Potential Fine Structure (EAPFS), have been applied to thin films of Ti/Si(111). Differences in techniques and analysis are outlined as well as criteria for analysis methods. Results show good agreement between these three techniques suggesting that the REELFS phase shifts obey pseudo-dipole selection rules as for EAPFS. Formation of titanium silicide is observed to occur at temperatures $\geq 500^\circ\text{C}$. Structures of the titanium silicide as measured by the EFS techniques are discussed.

Friday Morning - 10:50 a.m.

CHARACTERIZATION OF WINCHESTER DISK LUBRICANTS
USING ANGLE RESOLVED ESCA

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Recent improvements in ESCA instrumentation have led to a renewed interest in the use of angle-resolved ESCA to study thin ($<100\text{\AA}$) film structures such as the fluorocarbon lubricants on Winchester disks. Monitoring disk production with ESCA to assure optimal lubricant thickness is an essential part of disk manufacture. Assumptions are made about the morphology of the disk surface and the vacuum integrity of the lubricant. Angle-resolved ESCA and ESD reveal the shortcomings of these assumptions and establish the conditions required for valid lubricant thickness measurements. Angle-resolved ESCA also identifies any surface contamination covering the lubricant and suggests the molecular orientation of the lubricant.

Friday Morning - 11:10 a.m.

AN EVALUATION OF THE BREMSSTRAHLUNG
CONTRIBUTION TO SPECIMEN DAMAGE IN XPS
USING AUGER PEAK INTENSITIES*

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In conventional (i.e. non-monochromatic) x-ray sources used in x-ray photoelectron spectroscopy, the anode (usually Al or Mg) is bombarded by 10-15 keV electrons to produce the characteristic K α lines. Bremsstrahlung radiation is also produced in this process, which increases the total x-ray flux to the specimen. Since radiation damage to the specimen depends on the x-ray fluence used to generate an XPS spectrum, we were interested in determining what fraction of the total x-ray flux is comprised of bremsstrahlung, and what contribution this bremsstrahlung makes to specimen degradation.

A recent publication by Charles Wagner [Surf. Int. Anal. 6 (1984) 90] showed that this bremsstrahlung intensity is much less than is commonly believed by XPS users, and concluded that the contribution to specimen damage was only about 2% of the damage caused by the characteristic AlK $\alpha_{1,2}$ line. This result was based on the assumption that the main source of specimen damage is due to the photoelectrons and secondary electrons produced by x-ray absorption.

In this study, the ratio of the intensity of the main Auger peak to that of its associated photoelectron peak was measured for several materials using both a monochromatic Al K α x-ray source and a conventional Al K α source. The difference between these ratios for each material is due to the bremsstrahlung component from the conventional source. This difference was compared with that calculated from theoretical bremsstrahlung distributions and excitation cross-sections. Implications for specimen damage will be discussed.

* This work was sponsored by the Air Force Wright Aeronautical Laboratories, Materials Laboratory, Air Force Systems Command, United States Air Force, Wright-Patterson Air Force Base, Ohio 45433

Friday Morning - 11:30 a.m.

Scattering of Very Low Energy (<100 eV) Ne^+ Ions from Copper

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ABSTRACT

Determining the composition and position of atoms at the outermost surface of solids is one potential use of very low energy (<100 eV) ion surface scattering (ISS). In this application, ejection of surface atoms by the incident probe ions should be minimal. The present study describes (a) instrumental details and (b) design schemes of an operational Ion Scattering Mass Spectrometer (ISMS) which combines Ion Surface Scattering (ISS) with Secondary Ion Mass Spectrometry (SIMS). Magnetic analysis of secondary ions provides sufficient resolution of the sputtered and scattered ions at primary ion energies < 50 eV. Experimental results on non-destructive studies of the segregation of sulphur on a copper surface are discussed.

Friday Morning - 11:50 a.m.

SECTION V

CONCLUSIONS

The purpose for holding this symposium was to bring together basic research, applied research, and problem solving efforts all involving some type of surface analysis of Department of Defense interest into one meeting to try to improve communications and understanding of persons in these various endeavors. That a need for such a meeting exists seems, more and more, to be established from the many favorable comments received and from the continuing interest in this symposium.

The Office of Naval Research is to be commended for supporting this symposium.

Unclassified

ADA 169640

SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION Unclassified			1b. RESTRICTIVE MARKINGS		
2a. SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited		
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE					
4. PERFORMING ORGANIZATION REPORT NUMBER(S) UDR-TR-86-60			5. MONITORING ORGANIZATION REPORT NUMBER(S)		
6a. NAME OF PERFORMING ORGANIZATION University of Dayton		6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION		
6c. ADDRESS (City, State and ZIP Code) 300 College Park Ave. Dayton, OH 45469			7b. ADDRESS (City, State and ZIP Code)		
8a. NAME OF FUNDING/SPONSORING ORGANIZATION Office of Naval Research		8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER N00014-85-C-0118		
8c. ADDRESS (City, State and ZIP Code) 300 N. Quincy St. Arlington, VA 22217			10. SOURCE OF FUNDING NOS.		
11. TITLE (Include Security Classification) 7th Symposium on Applied Surface Analysis			PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.
12. PERSONAL AUTHOR(S) John T. Grant			WORK UNIT NO.		
13a. TYPE OF REPORT Final Report	13b. TIME COVERED FROM 3/18/85 TO 3/30/86	14. DATE OF REPORT (Yr., Mo., Day) July 1986		15. PAGE COUNT	
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB. GR.	surface analysis, adhesion, wear, thin films, thermionic emission, electronic devices, coatings, corrosion		
19. ABSTRACT (Continue on reverse if necessary and identify by block number)					
<p>The 7th Symposium on Applied Surface Analysis was held at the University of Maryland, 15-17 May 1985. This symposium was held to meet a need, namely to show the continuing transition between basic surface science research and applications of this research to areas of Department of Defense interest. Areas receiving special attention at this symposium were chemical bonding and reactions at metal-semiconductor interfaces, surface analysis and the tribological processes of ion implanted materials, microbeam analysis and laser ionization of sputtered neutrals. Other topics discussed included adsorption, adhesion, corrosion, wear, and thin films. Approximately 80 scientists active in the field of surface analysis participated in the symposium. Four scientists presented invited papers at the symposium. There were 26 contributed presentations. Laboratory tours of the University of Maryland, the National Bureau of Standards, and the Naval Research Laboratory</p> <p style="text-align: right;">CONTINUED</p>					
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS <input type="checkbox"/>			21. ABSTRACT SECURITY CLASSIFICATION Unclassified		
22a. NAME OF RESPONSIBLE INDIVIDUAL John T. Grant			22b. TELEPHONE NUMBER (Include Area Code) (513) 255-5125	22c. OFFICE SYMBOL UDRI	

19. ABSTRACT (Continued)

were conducted during the symposium. The proceedings of the symposium have been published in a special issue of the journal "Applied Surface Science" by North-Holland Publishing Co.

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